


FORM PTO-1390 (REV 10-95)		U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE	ATTORNEY'S DOCKET NUMBER MERCK 2320
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. §371			U.S. APPLICATION NO. (If known, see 37 CFR §1.5) 10/018827
INTERNATIONAL APPLICATION NO. PCT/EP00/05391	INTERNATIONAL FILING DATE 13 JUNE 2000	PRIORITY DATE CLAIMED 25 JUNE 1999	
TITLE OF INVENTION PIGMENT PREPARATION			
APPLICANT(S) FOR DO/EO/US GRIESSMANN, Carsten, et al.			
<p>Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:</p> <ol style="list-style-type: none"> <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under <u>35 U.S.C. §371</u>. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. §371. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(1). <input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. §371(c)(2)) <ol style="list-style-type: none"> <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau) <input checked="" type="checkbox"/> has been transmitted by the International Bureau. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US) <input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. §371(c)(2)). <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. §371(c)(3)) <ol style="list-style-type: none"> <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau) <input type="checkbox"/> have been transmitted by the International Bureau. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired. <input checked="" type="checkbox"/> have not been made and will not be made. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. §371(c)(3)). <input type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)). <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. §371(c)(5)). <p>Items 11. to 16. below concern document(s) or information included:</p> <ol style="list-style-type: none"> <input type="checkbox"/> An Information Disclosure Statement under 37 C.F.R. §§1.97 and 1.98 <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. §§3.28 and 3.31 is included. <input checked="" type="checkbox"/> A FIRST preliminary amendment. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment. <input type="checkbox"/> A substitute specification. <input type="checkbox"/> A change of power of attorney and/or address letter. <input type="checkbox"/> Other items or information: 			

U.S. APPLICATION NO (if known, see 37 CFR §1.5) <div style="font-size: 1.5em; font-weight: bold;">10/018827</div>		INTERNATIONAL APPLICATION NO PCT/EP00/05391		ATTORNEY'S DOCKET NUMBER MERCK 2320	
17. <input checked="" type="checkbox"/> The following fees are submitted: BASIC NATIONAL FEE (37 CFR §1.492 (a) (1) - (5)): Search Report has been prepared by the EPO or JPO..... \$890.00 International preliminary examination fee paid to USPTO (37 CFR §1.482)..... \$710.00 No international preliminary examination fee paid to USPTO (37 CFR §1.482) but international search fee paid to USPTO (37 CFR §1.445(a)(2))..... \$740.00 Neither international preliminary examination fee (37 CFR §1.482) nor international search fee (37 CFR §1.445(a)(2)) paid to USPTO..... \$1040.00 International preliminary examination fee paid to USPTO (37 CFR §1.482) and all claims satisfied provisions of PCT Article 33(2)-(4)..... \$100.00 <div style="text-align: right;">ENTER APPROPRIATE BASIC FEE AMOUNT =</div>				CALCULATIONS PTO USE ONLY	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 C.F.R. §1.492(e)).					
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	9 - 20 =	0	x \$ 18.00	\$0.00	
Independent claims	1 - 3 =	0	x \$ 84.00	\$0.00	
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ \$ 280.00		
TOTAL OF ABOVE CALCULATIONS =				\$890.00	
Reduction of 1/2 for filing by small entity, if applicable. A Verified Small Entity Statement must also be					
SUBTOTAL =				\$890.00	
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 C.F.R. §1.492(f)).					
TOTAL NATIONAL FEE =				\$890.00	
Fee for recording the enclosed assignment (37 C.F.R. §1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 C.F.R. §§3.28, 3.31). \$40.00 per property.					
TOTAL FEES ENCLOSED =				\$890.00	
				Amount to be refunded:	
				charged:	
a. <input checked="" type="checkbox"/> A check in the amount of <u>\$890.00</u> to cover the above fees is enclosed. b. <input type="checkbox"/> Please charge my Deposit Account No. <u>13-3402</u> in the amount of \$_____ to cover the above fees. A duplicate copy of this sheet is enclosed. c. <input checked="" type="checkbox"/> The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. <u>13-3402</u> . A duplicate copy of this sheet is enclosed					
<p>NOTE: Where an appropriate time limit under 37 C.F.R. §§1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. §1.137(a) or (b)) must be filed and granted to restore the application to pending status.</p> <p>SEND ALL CORRESPONDENCE TO Customer Number 23,599</p> <div style="display: flex; justify-content: space-between; align-items: flex-end;"> <div style="text-align: center;">  <div style="font-size: 1.2em; font-weight: bold;">23599</div> <div style="font-size: 0.8em;">PATENT TRADEMARK OFFICE</div> </div> <div style="text-align: right;"> <div style="border-bottom: 1px solid black; width: 150px; margin-bottom: 5px;"></div> <div style="font-size: 0.8em;">SIGNATURE</div> <div style="border-bottom: 1px solid black; width: 150px; margin-bottom: 5px;"></div> <div style="font-size: 0.8em;">NAME</div> <div style="border-bottom: 1px solid black; width: 150px; margin-bottom: 5px;"></div> <div style="font-size: 0.8em;">REGISTRATION NUMBER</div> </div> </div> <div style="display: flex; justify-content: space-between; margin-top: 10px;"> <div> Filed: 21 DECEMBER 2001 AJZ:kmo </div> <div style="text-align: right;"> <div style="border-bottom: 1px solid black; width: 150px; margin-bottom: 5px;"></div> <div style="font-size: 0.8em;">27,969</div> </div> </div>					

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JC03 Rec'd PCT/PTC 21 DEC 2001

IN THE UNITED STATES DESIGNATED/ELECTED OFFICE

International Application No. : PCT/EP00/05391
International Filing Date : 13 JUNE 2000
Priority Date(s) Claimed : 25 JUNE 1999
Applicant(s) (DO/EO/US) : GRIESSMANN, Carsten, et al.

Title: PIGMENT PREPARATION

PRELIMINARY AMENDMENT

Commissioner for Patents
Washington, D.C. 20231

SIR:

Prior to calculating the national fee, and prior to examination in the National Phase of the above-identified International application, please amend as follows:

IN THE CLAIMS:

1. (Amended) Non-dusting homogenous pigment preparation according to Claim 1, characterized in that the polyacrylate resin is an α -methylstyrene-modified polyacrylate.
2. (Amended) Non-dusting homogenous pigment preparation according to Claim 1, characterized in that it comprises, as additional components, defoamers, surface-active substances, wetting agents, anti-settling agents, levelling agents, siccatives and/or thixotropic agents.

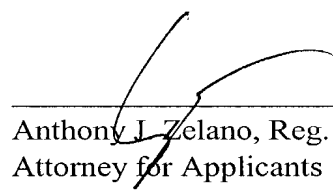
8. (Amended) Dry preparations produced from the non-dusting homogenous pigment preparation according to Claim 1 by shaping the dry preparations by tableting, briquetting, pelletizing, fluidized-bed granulation, granulation, spray-granulation or extrusion and freeing the dry preparations, if appropriate, from the solvent.

REMARKS

The purpose of this Preliminary Amendment is to eliminate multiple dependent claims in order to avoid the additional fee. Applicants reserve the right to reintroduce claims to canceled combined subject matter.

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attached pages are captioned "**Version With Markings to Show Changes Made**".

Respectfully submitted,



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Filed: 21 December

VERSION WITH MARKINGS TO SHOW CHANGES MADE

Claims 1, 2 and 8 were amended as follows:

9. (Amended) Non-dusting homogenous pigment preparation according to ~~one of~~ Claims 1 to 4, characterized in that the polyacrylate resin is an α -methylstyrene-modified polyacrylate.
10. (Amended) Non-dusting homogenous pigment preparation according to ~~one of~~ Claims 1 to 5, characterized in that it comprises, as additional components, defoamers, surface-active substances, wetting agents, anti-settling agents, levelling agents, siccatives and/or thixotropic agents.
8. (Amended) Dry preparations produced from the non-dusting homogenous pigment preparation according to ~~one of~~ Claims 1 to 7 by shaping the dry preparations by tableting, briquetting, pelletizing, fluidized-bed granulation, granulation, spray-granulation or extrusion and freeing the dry preparations, if appropriate, from the solvent.

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Pigment preparation

The invention relates to a non-dusting homogeneous pigment preparation notable in that it comprises at least 40% by weight of one or more effect pigments, 0.5-50% by weight of water and/or an organic solvent or solvent mixture, and 0.5-59.5% by weight of a styrene-modified polyacrylate resin having an acid number of >90 mg KOH/g based on the pigment. Pigment preparations of this kind are particularly suitable for producing dry preparations, such as granules, for example. The invention further relates to the production of the pigment preparation and the granules and to the use thereof for pigmenting printing inks.

In industrial processes, pigments are often not employed in the form of dry powders, since such powders produce dust, which results in heightened requirements in terms of workplace safety. In many cases, furthermore, when introducing powders into plastics, printing inks, industrial coatings, basecoat systems, etc., agglomeration of the pigment powder is observed. Homogeneous distribution of the pigment in the respective matrix is frequently difficult if not impossible to achieve.

Instead of the pigment powder, non-dusting pigment formulations or dry preparations are used. These are

- 30 - free-flowing powders, pearl lustre pigments being coated with polymers, as are described, for example, in DE-C-2603211,
- pigmented free-flowing powders having a low moisture content, as are known, for example, from 35 DE-A-4139993,
- pigmented powders having a relatively high moisture content, which owing to their flowable

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consistency are frequently referred to as pastes,

- pigment formulations in granule form, in which organic pigments are coated with a resin mixture, as disclosed, for example, in EP 0 897 956 A2, or
- dry preparations, such as pellets, granules as precursors for printing inks, as are known, for example, from EP 0 803 552 A1.

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Non-dusting homogeneous pigment preparations and dry preparations prepared from them are an industrial alternative to the dry or moistened powders provided that they meet the following general conditions:

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- flowable consistency
- minimal dilatancy
- maximum pigment content

20 The components of the preparation/paste, or of the dry preparations, should be chosen such that the formulation is harmonized very effectively with the other components of the respective coating system and is readily homogeneously distributed following its

25 introduction.

In addition to good compatibility with the other constituents of the coating system, pigment preparations, and the dry preparations produced from them, are required to result in high stability in the coating systems, i.e., they must not tend towards phase separation, or, where this cannot be entirely prevented owing to the low viscosity, the coating systems must be able to be returned easily to a homogeneous state, i.e., the settled sediment must be easy to reagitate. This requirement is particularly important in the case of platelet-shaped pigments, since owing to their structure such pigments have a tendency to undergo "caking" on phase separation and are difficult to

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reagitate. Pigments based on platelet-shaped substrates give rise to handling problems insofar as, owing to the size and density of the pigments, they readily settle and may then cake together to form a very firm sediment
5 cake. This cake is generally difficult to reagitate. This is particularly so in connection with the storage of varnishes, paints and printing inks, as well as in connection with their processing.

10 For this reason, numerous methods, inter alia, have been developed in order to solve the problem of the incorporation and handling of platelet-shaped pigments in coating compositions. Reagitation can be facilitated by adding to the coating compositions additives which
15 alternatively bring about controlled flocculation (house of cards effect), pseudoplastic and/or thixotropic behaviour, or steric and/or electrostatic repulsion of the pigments. However, these additives may have an adverse effect on the quality of the coating.
20 In particular, the brilliance in the case of effect pigments, and the uniformity of the coating, may be impaired.

In addition, homogeneous, stable distribution of the
25 redispersants in the pearl lustre pigment powder is difficult to achieve, and/or the redispersant loses some of its activity in the course of mixing.

The effect pigment formulations developed to date for
30 use in coating systems, with a pigment content >30% by weight, frequently do not go far enough towards meeting the requirements described above, especially since they have a tendency towards agglomeration and shear thickening.

35 The object of the present invention was therefore to provide pigment preparations, especially those in the form of formulations or pastes and dry preparations, which simultaneously can be used very effectively in

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effect pigment, such as a pearl lustre pigment, for example, are distinguished by a high glass transition temperature. This fact ensures a blocking-resistant formulation of the paints, varnishes, printing inks and overprint varnishes produced therefrom. Furthermore, the granules of the invention are distinguished by excellent compatibility with the customary resins and additives.

Moreover, the products of the invention are non-dusting, readily free-flowing, can be incorporated rapidly into commercially available binder systems, and are compatible therein. In particular, the products are compatible with aqueous and solventborne systems and in some cases compatible in solvent-free printing ink and coating systems as well. The paints and varnishes produced from the dry preparation, e.g. granules, are suitable for gravure, flexographic and screenprinting, offset overprint varnishes, overprint varnishes (OPVs), and also the various coating systems from the industrial coatings and automotive sector. They are also suitable for colouring plastics.

The pigment preparation of the invention contains preferably 40-98% by weight of effect pigments, especially 45-95% by weight, based on the preparation. Very particular preference is given to pigment preparations having an effect pigment content of more than 50% by weight. The percentages by weight are based in each case on the moist, i.e. undried, pigment preparation.

Effect pigments used are pigments based on platelet-shaped, transparent or semi-transparent substrates comprising, for example, sheet silicates, such as mica, synthetic mica, platelet-shaped iron oxide, SiO_2 flakes, TiO_2 flakes, graphite flakes, Fe_2O_3 flakes, Al_2O_3 flakes, glass flakes, holographic pigments, talc, sericite, kaolin, or other silicatic materials coated

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The pigment preparations of the invention may comprise one or more effect pigments. In many cases it is possible by using at least two different effect pigments to obtain special colour effects and lustre effects. Preferred pigment preparations comprise one or more effect pigments based on mica and/or SiO₂ flakes. Blends with up to 10% by weight of organic and inorganic pigments, based on the pigment preparation, are also possible.

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In particular, the addition of one or more dyes and/or organic pigments in dispersed form leads to special colour effects. It is also possible to add substances and particles (tracers) which enable the product to be identified (product protection).

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As a mandatory component, the pigment preparation of the invention comprises a styrene-modified polyacrylate resin having an acid number >90 mg KOH/g in amounts of from 0.5 to 59.5% by weight, preferably from 5 to 40% by weight, in particular from 10 to 30% by weight. Particular preference is given to styrene-modified polyacrylate resins having an acid number of >100, in particular of >150.

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The styrene-modified polyacrylates used have an average molecular weight of from 500 to 200,000, preferably from 1000 to 100,000, in particular from 1000 to 50,000. In the case of otherwise identical properties, the polymers having a low molecular weight are preferred, since they generally have a smaller thickening effect in the coating systems.

30

The styrene-modified polyacrylates are prepared by free-radical addition polymerization, especially copolymerization or terpolymerization, in the course of which it is possible to incorporate saturated and unsaturated alcohols and also other functionalizations in broad mass proportions. In particular, polyethylene

35

In particular, it is also possible to add substances which accelerate or assist the breakdown and dissolution of the pigment granules, examples being bulky spherical particles, such as hollow beads, hemispherical beads, swellable substances with a strong swelling power, substances which undergo controlled breakdown and in doing so evolve gases, and/or substances having very high solubility.

The acid number of the polyacrylate in conjunction with the neutralizing agent gives the resins water solubility. Water solubility in accordance with the present invention is on the one hand important in connection with the preparation process, in order to permit an aqueous route, while on the other hand the water solubility is a prerequisite for use in aqueous and waterborne printing ink systems (compatibility).

In general, the pigment preparation of the invention contains from 0 to 10% by weight of a neutralizing

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agent, preferably from 1 to 7% by weight, in particular from 1 to 5% by weight, based on the pigment preparation.

If necessary, the addition of a redispersing aid in the form of bulky particles, such as fibres or spherical particles, for example, prevents the effect pigments treated in accordance with the process of the invention from lying on top of one another to a notable extent as a result of steric repulsion and so exerting strong adhesion. The result of this is that

1. the preparations of the invention are more stable, and
2. owing to the introduction of the redispersing aid into the paint or varnish system by way of the pigment preparation, the effect pigments settle more slowly, in some cases very much more slowly, in paint and varnish systems;

in all cases, however, the sediment is less hard, and no problems occur when the sediment is reagitated.

The redispersant is preferably used in amounts of from 0 to 5% by weight, in particular from 0.05 to 3% by weight, based on the pigment preparation. All organic and inorganic fibres known to the person skilled in the art and having a fibre length of 0.1-20 μm can be used. Particularly suitable particles are all synthetic fibres, made for example from polyethylene, polyacrylates, polypropylene, polyamides, cellulose fibres, inorganic fibres, preferably silicon compounds, glass fibres, and also, in particular, the condensation products of modified isocyanates and mono- and diamines.

These condensation products, which are diurea derivatives and aminoureas having urethane groups, are known as thixotropic agents and, together with a

Redispersing aids which can be used are all those diurea derivatives and urethane compounds known to the person skilled in the art, as are described, for example, in EP 0 198 519, DE 18 05 693.4, and in Organic Coatings: Science and Technology, A. Heenriga, P.J.G. von Hemsbergen, pp. 201-222, New York 1983.

Suitable spherical materials are, in particular, hollow glass, wax or polymer beads made from vinyl resins, nylon, silicone, epoxy resins, olefin resins and polystyrenes, and from inorganic materials, such as TiO_2 , SiO_2 or ZrO_2 , for example. Preference is given to the use of hollow beads, and also solid beads, having a particle size of from 0.05 to 150 μm . With particular preference, hollow glass, wax or polymer beads are used in the pigment preparation of the invention.

Spherical particles based on SiO_2 in a particle range of 3-10 μm are known, for example, as materials for high-performance liquid chromatography and are sold, for example, as LiChrospher® by Merck KGaA, Darmstadt, FRG. Such materials are preferably used in monodisperse form; that is, with a substantially uniform particle size. Monodisperse spherical particles of this type based on SiO_2 , TiO_2 and ZrO_2 are known. Monodisperse SiO_2 , for example, can be prepared in accordance with DE 36 16 133. Hollow glass beads are sold, for example, under the trade name Q-CEL by PQ Corporation, USA, or Scotchlite by 3M, Frankfurt, FRG.

In addition, the pigment preparation of the invention
35 may comprise surface-active substances, such as
alkylsilanes, for example, which may also contain a
further functional group, or unsaturated or saturated
fatty acids or fluorosurfactants. Particular preference
is given to the use of silane compounds of the formula

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(C_nH_{2n+1})Si(OC_mH_{2m+1})₃, where n is 1-30 and m is 1-10, as surface-active substances. Examples of suitable silane compounds are n-hexyldecyltriethoxysilane and n-octyldecyltriethoxysilane (Si 116 and Si 118, respectively, from Degussa AG, Frankfurt, FRG), and also the corresponding fluoroalkylsilanes.

In addition to the silane, the pigment preparation preferably further comprises a surfactant or a fatty acid. The surface-active reagent may also comprise a mixture of silane, fatty acids and/or surfactants. The pigment preparations can contain from 0.1 to 5% by weight, preferably from 0.2 to 3% by weight and, in particular, from 0.5 to 2% by weight of surface-active substances, based on the preparation.

Further surface-active substances which may be used are the saturated and unsaturated fatty acids, such as capronic acid, caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, stearic acid, oleic acid, linoleic acid, and also fatty acid mixtures, for example.

The pigment preparation of the invention contains from 0.5 to 50% by weight of water and/or an organic solvent or solvent mixture, preferably from 1 to 45% by weight, in particular from 1 to 40% by weight, based on the preparation.

The solvent component in the pigment preparation of the invention must be properly matched to the resin having an acid number >90 mg KOH/g. For the preparation, water and all organic solvents can be used depending on the polyacrylate resin employed. Examples of suitable solvents are aromatic solvents such as toluenes, benzines, xylenes, hydrocarbons and also esters, cyclohexane, furans, dioxanes, chlorinated hydrocarbons, long-chain amines, vegetable oils, monohydric aliphatic alcohols, such as those having 2

15 The pigment preparation of the invention is produced in a simple manner by adding the polyacrylate resin, with or without water and/or an organic solvent or solvent mixture, a modifier and, if desired, further additives, simultaneously or in succession to the effect pigment
20 or effect pigment mixture and subjecting the resulting mixture to gentle homogenization in a mixing apparatus.

During or after its production, further customary additives may be added to the pigment preparation of the invention, examples being defoamers, wetting agents, anti-settling agents, levelling agents, siccatives and thixotropic agents. These are auxiliaries which are customary in the coatings industry and which may be present in the pigment preparation of the invention in an amount of from 0 to 10% by weight. Mention is made here in particular of

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succinate derivatives, examples being those as sold by Henkel under the brand name Hydropalat 875.

The resultant moist pigment preparation of the invention is a homogeneous powder, or readily flowable pastes, having a relatively high effect pigment content. Because of the particularly high level of compatibility of the styrene-modified polyacrylate resin, the pigment preparation of the invention is likewise thoroughly compatible with commercially customary systems. Further features which distinguish the preparation are its freedom from dust, ready dispersibility and ready redispersibility, and it is therefore markedly superior to conventional pigment preparations.

In order to produce dry preparations, the pigment preparation of the invention is extruded or, by other methods known to the person skilled in the art, compressed into a compact particulate form by means, for example, of tabletting, briquetting, pelletizing, granulating, spray-granulating, fluidized-bed granulating, or extrusion, and may then be dried. The drying procedure generally takes place at temperatures from 20°C to 150°C, preferably at from 60 to 120°C, and can if desired be carried out under reduced pressure. The dry preparations, such as granules, for example, have a residual moisture content of not more than 5% by weight, preferably <3% by weight. The granules have pigment contents of >70% by weight, preferably of >80% by weight, based on the granules. Finally, the dry preparation is, if desired, classified. The dried granules obtained in this way have positive properties which are further increased relative to the water- and/or solvent-containing preparations: the absence of liquid components increases the compatibility. Dry granules in particular also, surprisingly, showed no disadvantages in terms of wetting. On the contrary, when added to an aqueous binder or to a binder based on

The improved deagglomeration of the dry preparations when incorporated into a binder, such as into a printing ink, is evident even with small amounts of spherical particles in the pigment preparation. For instance, even when using granules having a spherical particle content of 0.5% by weight based on the dry preparation, the dissolution rate is markedly increased

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and the stability of the printing ink is attained more rapidly (viscosity/hue).

The dry preparations are non-dusting, readily free-flowing, can be incorporated rapidly into commercially available binder systems, and are compatible therein. In particular, the products are compatible not only with aqueous and solventborne but also with solvent-free printing inks and coating systems.

As a paste or as a dry preparation, the preparation of the invention can be used for diverse applications. It is preferably used in coating systems from the sectors of printing, especially overprint varnishing, offset overprint varnishing, and gravure, flexographic and screenprinting. With particular preference, the preparation or the dry preparation produced from it is applied as a precursor for coating compositions on any desired substrate materials, examples being metals such as iron, steel, aluminium, copper, bronze; plastic; brass and also metal foils, and also glass, ceramic and concrete, and also on wood, e.g. furniture, clay, textile, paper, packaging materials, e.g. plastic containers, films or boards, or on other materials for decorative and/or protective purposes.

The invention therefore also provides for the use of the pigment preparation in formulations such as paints, varnishes, printing inks and plastics, and also automotive finishes, powder coating materials and coating compositions in general.

The examples which follow are intended to illustrate the invention without, however, restricting it.

Example 1

679 g of Joncryl® HPD 671 resin solution (26% strength solution of a styrene-modified polyacrylate resin

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consisting of 80 g of AMP 75, 176.5 g of Joncryl® HPD 671 having an acid number of 214 and a molecular weight of 17250, and 922.5 g of DI water) are mixed homogeneously in a R02 mixer from Eirich with 1000 g of
 5 Iriodin® 123 (TiO₂ mica pigment with a particle size of 5-20 µm from Merck KGaA). The moistened mixture is granulated on a TR01 granulating plate from Eirich. The moist, granulated mixture is dried at 80°C and 100 mbar. The dried granules are passed first over a
 10 sieve having a mesh size of 2 mm and then over a sieve having a mesh size of 200 µm.

The product is abrasion resistant, dimensionally stable, and compatible with the common aqueous binder
 15 Senolith high-gloss varnish 350081 (offset overprint varnish based on a styrene-acrylate dispersion, containing a sodium salt of sulfosuccinic acid as modifier) from Weilburger Lackfabrik.

20 Example 2

A solution is prepared from 69.8 g of Joncryl® 682 granulating resin (styrene-modified polyacrylate resin having a molecular weight of 1800 from Johnson
 25 Polymers), 287.4 g of DI water, 5.4 g of 25% strength ammonia solution, 37.0 g of AMP-75 (2-amino-2-methyl-1-propanol), and 14.0 g of polyethylene glycol 2000 (modifier) and this solution is mixed homogeneously in a 4-paddle mixer with 586.4 g of Iriodin® 100 (TiO₂ mica pigment of particle size 10-60 µm from Merck KGaA).
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The preparation produced in this way is granulated on a TR01 granulating plate from Eirich. The granules are dried at 100°C. In order to separate the coarse
 35 fraction and fine fraction from the desired fraction, the dry granules are first passed over a coarse sieve (upper particle limit 1.6 mm) and then the dust and the fine fraction is separated off over a fine sieve (particle size less than 0.4 mm). The granules prepared

The granules prepared in this way are abrasion resistant, dimensionally stable, and readily compatible with Senolith high-gloss varnish 350081 (offset overprint varnish based on a styrene-acrylate dispersion, containing a sodium salt of sulfosuccinic acid as modifier) from Weilburger Lackfabrik.

Example 4

A solution is prepared from 69.8 g of Joncryn[®] 682
granulating resin (styrene-modified polyacrylate resin
5 having a molecular weight of 1800 from Johnson
Polymers), 287.4 g of DI water, 5.4 g of 25% strength
ammonia solution, 37.0 g of AMP-75 (2-amino-2-methyl-1-
propanol), and 14.0 g of polyethylene glycol 2000
(modifier) and this solution is mixed homogeneously in
10 a 4-paddle mixer with 586.4 g of Fe₂O₃-coated SiO₂
flakes of particle size 5-40 µm (from Merck KGaA).

The preparation produced in this way is granulated on a
TR01 granulating plate from Eirich. The granules are
15 dried at 100°C.

In order to separate the coarse fraction and fine
fraction from the desired fraction, the dry granules
are first passed over a coarse sieve (upper particle
20 limit 1.6 mm) and then the dust and the fine fraction
is separated off over a fine sieve (particle size less
than 0.4 mm).

The granules prepared in this way are abrasion
25 resistant, dimensionally stable, and readily compatible
with the common solvent-free aqueous binder/coating
systems, such as, for example, Senolith high-gloss
varnish 350081 from Weilburger Lackfabrik from the
printing sector.

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Example 5

413.6 g of a 16.9% strength Joncryn[®] 682 solution
(solution consisting of 287.4 g of water, 14 g of
35 polyethylene glycol 2000, 42.2 g of 2-amino-2-
methylpropanol (75% strength) and 69.8 g of Joncryn[®]
682) are mixed homogeneously in a R02 mixer from Eirich
with 586.4 g of Iriodin[®] 100 (TiO₂ mica pigment with a
particle size of 10-60 µm from Merck KGaA). The

- 20 -

moistened mixture is granulated on a TR01 granulating plate from Eirich. The moist, granulated mixture is dried at 80°C and 100 mbar. The dried granules are passed first over a sieve with a mesh size of 2 mm and then over a sieve with a mesh size of 200 µm. The product is abrasion resistant and dimensionally stable and readily compatible with the common aqueous binder/varnish systems, such as, for example, Senolith high-gloss varnish 350081 from Weilburger Lackfabrik from the printing sector.

Comparative example (EP 0 803 552 A1)

Granules comprising pearl lustre pigment (Iriodin® 123) and polyacrylate (not styrene-modified)

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643 g of Neocryl B817 (17.5% strength neutralized solution in AMP 75 of a MMA/EA copolymer in water, having an acid number of 59, a T_g of 65°C and an average molecular weight of 20,000, from Zeneca Resins, Waalwijk, Netherlands) and 1000 g of Iriodin® 123 (TiO₂ mica pigment of particle size 5-20 µm from Merck KGaA) are mixed homogeneously in a mixer. The moistened mixture is granulated on a TR01 granulating plate from Eirich. The moist, granulated mixture is dried at 80°C and 100 mbar. The dried granules are passed first over a sieve with a mesh size of 2 mm and then over a sieve with a mesh size of 200 µm.

The product is stable to abrasion and dimensionally stable. In comparison to Examples 1-6, it disperses much more slowly in the test binder Senolith high-gloss varnish 350081 from Weilburger Lackfabrik and in contrast to styrene-modified polyacrylate resin having acid numbers >90 it shows only retarded dissolution in conventional and aqueous binders and overprint varnishes. At the same time, increased tendency to foam was noted when the Neocryl B 817 solid resin was being hydrolysed.

[illegible]

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DECLARATION FOR PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name,

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

PIGMENT PREPARATION

the specification of which

☐ is attached hereto

☒ was filed on 13 JUNE 2000 as United States Application Number or PCT International Application Number PCT/EP00/05391 and (if applicable) was amended on _____

I hereby authorize our attorneys to insert the serial number assigned to this application.

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56.

I hereby claim foreign priority benefits under 35 U.S.C. §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

PRIOR FOREIGN/PCT APPLICATION(S) AND ANY PRIORITY CLAIMS UNDER 35 USC §119			
APPLICATION NO.	COUNTRY	DAY/MONTH/YEAR FILED	PRIORITY CLAIMED
199 29 378.3	GERMANY	25 JUNE 1999	YES

I hereby claim the benefit under 35 U.S.C. §119(e) of any United States provisional application(s) listed below.

PROVISIONAL APPLICATION(S) UNDER 35 U.S.C. §119(e)	
APPLICATION NUMBER	FILING DATE

I hereby claim the benefit under 35 U.S.C. §120 of any United States application, or §365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. §112.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR §1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application

PRIOR U.S./PCT INTERNATIONAL APPLICATION(S) DESIGNATED FOR BENEFIT UNDER 37 U.S.C. §120		
APPLICATION NO.	FILING DATE	STATUS — PATENTED, PENDING, ABANDONED

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected herewith. I William Millen (19,544); John L. White (17,746); Anthony J. Zelano (27,969); Al E. J. Branigan (20,565); John R. Moses (24,983); Harry B. Shubin (32,004); Brian P. Heaney (32,542); Richard J. Traver (30,595); John A. Sopp (33,103); Richard M. Lebovitz (37,067); John H. Thomas (33,460); Catherine M. Joyce (40,668); Nan J. Axelrod (44,014); James T. Moore (35,619); James E. Ruland (37,432); Jennifer J. Branigan (40,921) and Robert McCarthy (46,044)

23599

PATENT TRADEMARK OFFICE

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